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U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION 10
1200 SIXTH AVENUE
SEATTLE, WASHINGTON 98101

AUG 17 1987

REPLY TO
ATTN OF: ES-096

MEMORANDUM

SUBJECT: Review of Butyltin Analysis in Sediment Samples

FROM: Raleigh Farlow, Chemist
Regional Quality Assurance Management Office

TO: Mike Matta, Environmental Scientist
Engineering and Investigation Section

At your request, a review has been completed of two documents; "Measuring Tri-n-Butyltin in Salmon by Atomic Absorption: Analysis With and Without Gas Chromatorgraphy" and "Analysis of Butyltins in Sediment Samples Provided by the Environmental Protection Agency (7/20/87)", both authored by Jeffrey Short who is with Auke Bay Laboratory. Mr. Short was contacted by telephone (8/12/87) for clarification of a few matters.

The first referenced document cites methods inwhich some of the techniques used were common to the method employed for sediment analysis. The methods cited in the two separate documents are similar but not the same.

The sediment analysis was conducted with sufficient quality control to yield a reasonable assessment of data quality. Reagent/procedural blanks, method recoveries, matrix recoveries, and sample duplicates were measured/analyzed for mono-, di- and tri-butyltin. Duplicate splits and matrix spikes were accomplished after preliminary sample preparation that included sample freeze-drying. Entire procedural replication and potential for analyte loss during the freeze-drying step were not determined.

Summary of results of the analysis of sediments and methods blanks generated from tables 1 and 3 in the sediment analysis document, are summarized as sample means and associated variabilities (expressed as RPD and CV for relative percent difference and coefficient of variation, respectively)

	T B T		D B T		M B T	
	Mean (ppm)	RPD	Mean (ppm)	RPD	Mean (ppm)	RPD
87060040	8.46	37	3.01	36	.84	67
87060041	12.28	.2	4.94	28	.74	17
87060042	.85	56	.29	28	.10	10
87060043	2.08	68	.54	2	.21	34
87060044	8.02	31	1.94	48	.51	82
87060045	2.62	10	.94	6	.18	40
		CV(%)		CV(%)		CV(%)
Method/reagent blank	.24	71	.2	–	.18	118
Matrix spike recoveries(%)	103	25	108	16	99	29

Reagent blank data was generated as the mean for each parameter in table 3 (in ug) divided by 0.2 gm as the average sample size; yielding an equivalent method blank for sediments. The coefficient of variation in the above table can be approximated to RPD determined from duplicate analyses.

Generally, there is concurrence with the data evaluation discussion at the end of the sediment analysis report. Analytical variability ranges from 0.2% to 82% and is apparently random. Much of the variability may be due to the small sediment sample size (0.2 gm.); where the method is capable of accepting up to 10 gm. solid samples. Note that the reagent blank results and small sizes control the method detection limits at 0.2-0.5 ppm. The recoveries are apparently good, but the variability as determined for this work would suggest that the quantitative data be considered as approximate or estimates (generally, chemistry data with > 25-30% variability are considered acceptable as approximate or estimated levels). The data should be usable for most purposes, however determination of trends should only be attempted with caution since the analytical variability is significant and a limited number of samples were collected and analyzed.

If you should have any questions or concern, please give me a call at 2-1193.